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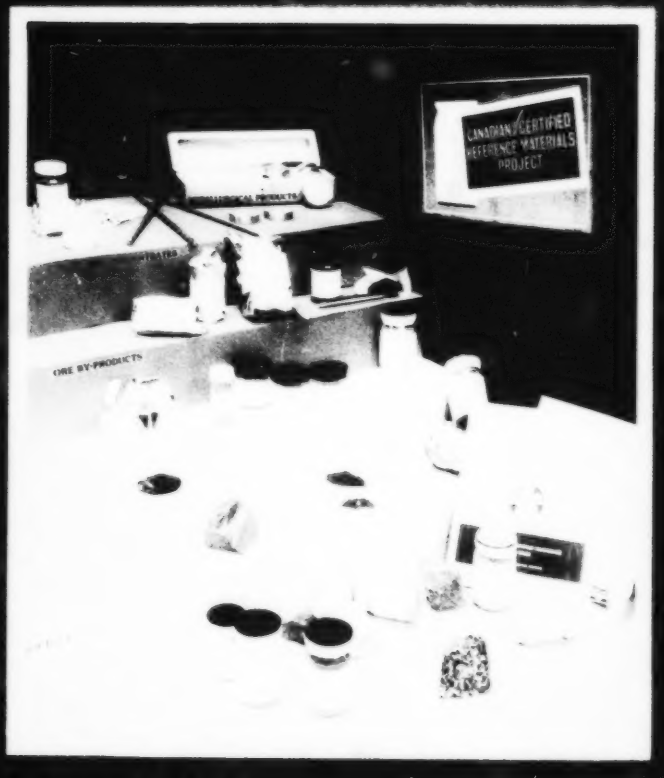
CUP–2: A CERTIFIED URANIUM ORE CONCENTRATE

J.L. Dalton and W.S. Bowman



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CUP-2: A CERTIFIED URANIUM ORE CONCENTRATE

J.L. Dalton* and W.S. Bowman**

SYNOPSIS

Uranium Ore Concentrate CUP-2 was analyzed in an interlaboratory program by 16 different laboratories from three different countries for the uranium content and many impurities. A member of the Analytical Subcommittee of the Canadian Uranium Producers Metallurgical Committee donated the material that was used in the project. The Canadian Certified Reference Materials Project carried out the blending, bottling and administration of the interlaboratory program.

The certified value for uranium in CUP-2 is 75.42 wt %, and recommended values are supplied for some 16 analytes.

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CUP–2 : CONCENTRÉ DE MINERAI D'URANIUM CERTIFIÉ

J.L. Dalton* et W.S. Bowman**

RÉSUMÉ

Le concentré de minerai d'uranium CUP–2 a été analysé dans un programme inter–laboratoires par 16 différents laboratoires de trois différents pays qui en ont dosé l'uranium et plusieurs impuretés. Un membre du sous–comité analytique du Comité de métallurgie des producteurs d'uranium canadiens a fourni le matériau utilisé dans le projet. La préparation, l'embouteillage et l'administration du programme inter–laboratoires ont été réalisés dans le cadre du Projet des matériaux de référence certifiés canadiens.

La teneur certifiée en uranium du CUP–2 est de 75,42 % en poids, et des valeurs recommandées sont fournies pour quelque 16 analyses.

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INTRODUCTION

The Analytical Subcommittee of the Canadian Uranium Producers Metallurgical Committee felt that it would be highly desirable to have a uranium ore concentrate available as a reference material. As far as is known, no commercially available material exists. Such a project would have to be a joint effort between the Analytical Subcommittee and CANMET under the Canadian Certified Reference Materials Project (CCRMP). The two groups decided that the Subcommittee would donate the material and its eight member laboratories would provide analytical services. CCRMP would homogenize and bottle the material and be responsible for the interlaboratory analytical program.

Uranium ore concentrate (UOC) is a generic term that is applied to a uranium precipitate produced at a minesite. The precipitate is, in almost all cases, an ammonium or magnesium diuranate $(NH_4)_2U_2O_7$ or $Mg_2U_2O_7$. According to ASTM Designation C967–81 (1), UOC must contain at least 65 wt % uranium, and the isotopic content must be that of naturally occurring uranium. The composition of UOC varies with both the mineralogy of the orebody and the milling process used. The Designation includes concentration specifications for some 19 analytes that have both penalty and rejection limits.

Twenty–five kg of UOC, labelled CUP–2, was received from the Blind River refinery of Eldorado Resources Ltd. in the summer of 1986. The material was blended, checked for homogeneity by X–ray fluorescence analysis, bottled in 50–g units and then distributed for analysis by late 1986. The material was to be analyzed for as many impurities as possible, as well as for the uranium content. The homogeneity test is described in Appendix A.

INTERLABORATORY PROGRAM

It was immediately apparent that there would not be enough laboratories to certify constituents of CUP–2 if only members of the Analytical Subcommittee participated in the interlaboratory program. Accordingly, CCRMP engaged commercial laboratories to participate in the program. In addition to these six laboratories and those of the Subcommittee, two American and one Australian volunteer laboratories participated in the project.

Each participating laboratory (listed in Appendix B) was assigned a confidential code number that bears no relation to its alphabetical order.

Each laboratory was requested to contribute five replicate results for each analyte. The uranium was to be reported on a dry basis; all other analytes were to be reported on an "as received" basis. The choice of analytical methodology was left to the expertise of the participants, bearing in mind the end use of the results. Compositional reference materials were to be avoided for calibration purposes to minimize the propagation of un-

certainties. The results received from the laboratories are detailed in Appendix D. If laboratories submitted results by two different methods, the two sets were considered statistically independent.

STATISTICAL TREATMENT OF ANALYTICAL RESULTS

Detection of Outliers

Cochran's test was used to identify data sets with abnormally high coefficients of variation (CV) (2). These sets were then examined individually by Dixon's test to determine whether the high variance was caused by one or more values that were remote from the mean of the set (2). Such values were excluded, and the coefficients of variation were recalculated. Sets continuing to have abnormally high CVs were not used for further computations.

Data sets whose means differed by more than twice the overall standard deviation from the grand mean were also excluded from calculations of recommended values and other statistics. All outliers are identified in Appendix D, where all the analytical data for the interlaboratory program are listed.

Estimation of Consensus Values and 95% Confidence Limits

A one–way analysis of variance technique was used to estimate the consensus values and variance. This approach considers the results of the described certification program to be only one sampling out of a universal set of results. The analytical data were assumed to fit the model (3):

$$x_{ij} = \mu + y_i + e_{ij}$$

where x_{ij} = the j^{th} result in set i,

μ = the true consensus value

y_i = the discrepancy between the mean of the results in the set i ($\bar{x}_i$.) and μ, and

e_{ij} = the discrepancy between x_{ij} and ($\bar{x}_i$)

It is assumed that both y_i and e_{ij} are normally distributed with means of zero and variances of ω^2 and σ^2, respectively. The significance of ω^2 is detected by comparing the ratio of between–set mean squares to within–set mean squares, with the F statistic at the 95% confidence level and with the appropriate degrees of freedom.

The consensus value of the assumed model is estimated by the overall mean $\bar{x}$..

$$\bar{x}.. = \sum_{i}^{k} \sum_{j}^{n_i} x_{ij} \Big/ \sum_{i}^{k} n_i$$

where n_i = the number of results in set i, and

k = the number of sets.

The value of σ^2 is estimated by the s_1^2, which is given by

$$s_1^2 = \sum_i^k \sum_j^{n_i} (x_{ij} - \bar{x}_{i.})^2 \Big/ \sum_i^k n_i - k$$

where $\bar{x}_{i.} = \sum_j^{n_i} x_{ij}/n_i$.

The value of ω^2 is estimated by

$$\omega^2 = (s_2^2 - s_1^2) \Big/ \frac{1}{k-1} \left(\sum_i^k n_i - \sum_i^k n_i^2 / \sum_i^k n_i \right)$$

where

$$s_2^2 = \sum_i^k n_i (\bar{x}_{i.} - \bar{x}_{..})^2 \Big/ k - 1.$$

The variance of the overall mean is given by

$$V[\bar{x}_{..}] = \left(\sum_i^k n_i^2 / (\sum_i^k n_i)^2 \right) \omega^2 + \left(1 / \sum_i^k n_i \right) \sigma^2$$

and the 95% confidence limits for $\bar{x}_{..}$ are

$$\bar{x}_{..} \pm t_{0.975,\,(k-1)} \sqrt{V[\bar{x}_{..}]}$$

where $t_{0.975,\,(k-1)}$ is Student's t statistic for $k-1$ degrees of freedom at a level of significance of 0.025.

It should be noted that 95% confidence limits denote that if the certification program were performed 100 times, the overall mean in 95 instances would fall within the prescribed limits.

The average within-set standard deviation, σ_A, is a measure of the average within-bottle precision as determined by the analytical methods used. The implication, therefore, is that a laboratory using a method of average or better reproducibility should obtain individual results for a given certified element with a precision that is at least comparable to the reported value of σ_A. The results of the statistical analysis of the analytical data are given in Appendix C.

Criterion for Certification

The ratio of the between-laboratory to the within-laboratory standard deviation, σ_B/σ_A, where

$$\sigma_B = \sqrt{\sum_i^k (\bar{x}_i - \bar{x}_{..})^2/(k-1)}$$

is a measure of the quality of the certification data for the reference materials of CCRMP (4). For a value obtained by the consensus of interlaboratory measurements to be certifiable, it must meet the criteria adopted by the CCRMP, that is: (1) there shall be at least 10 acceptable, independent sets of measurements, and (2) the fraction of sets, RP, necessarily rejected to obtain $\sigma_B/\sigma_A \leq 3$ shall be less than 15%.

DISCUSSION

The certified value for uranium and the recommended values for 16 impurity elements in CUP-2 are given in Tables 1 and 2. The distribution of uranium results is shown in Figure 1.

The results for uranium meet the criteria for certification by CCRMP. The test value of σ_B/σ_A is 2.4 when RP = 0%, and the ratio drops to 2.0

when RP = 14%. Thus, CUP–2 meets the more stringent criteria for certification of uranium proposed by Steger, based on the observed advanced state of consensus in uranium analyses (4).

Table 1 – Certified value for uranium in CUP–2

Mass %		
U		75.42
95% Confidence limits	Low	75.25
	High	75.59

Table 2 – Recommended values for elements/ constituents in CUP–2

Mass %			
Moisture	2.94	B	0.0051*
S	0.80	Fe	0.311
Si	0.17*	Ni	0.0029
Ti	0.019	Na	0.459
Ca	0.62	Mo	0.069
V	0.066*	Mg	0.229
As	0.035	K	0.11*
Zr	0.044*	P	0.030*

*provisionally recommended

For the remaining (impurity) constituents, the quality of consensus was sufficient for certification of sodium and magnesium only. The fact that there is a considerable variation in the analytical data for the impurity elements is a commentary on the present state of laboratory agreement in the industry for the analysis of UOC. As far as is known, there is no other similar material available that has been analyzed for both uranium content and impurities. Thus, CUP–2 should serve as a valuable reference material for both the Canadian and worldwide industry in quality control programs, method development projects and characterization studies.

Recommended values for 8 impurity constituents obtained from four or more independent sets of results are presented in Table 2.

An arbitrary distinction was made between recommended values and those identified in Table 2 as "provisionally recommended" values. The former are based on at least nine accepted sets (44 to 69 individual determinations) and meet an arbitrary test of quality of consensus $\sigma_B/\sigma_A \leq 4$ for RP $\leq$ 20%. Provisionally recommended values either are based on fewer than nine sets (boron, phosphorus and zirconium) or exhibit a lower quality of consensus.

The statistical parameters associated with the values of Tables 1 and 2 are summarized in Appendix C. These include the within–laboratory and between–laboratory standard deviations, S_{rc} (or σ) and S_{Lc} (or ω), respectively. These parameters have been proposed for use in assessing the precision and accuracy of a test method using a reference material, in comparison with methods accepted in the interlaboratory certification program (5).

A desirable feature of any interlaboratory program is to have the same element determined by several

different techniques. However, in this inter-laboratory program, most of the metal impurity determinations were made by ICP, DCP and AA techniques, but other techniques were used to strengthen the validity of the optical methods. X-ray fluorescence was used for the determination of titanium, calcium, vanadium and iron in addition to uranium. Sulphur was analyzed by both combustion and gravimetric techniques. Spectrophotometry was used for the determination of arsenic, boron and iron. Many elements listed in Appendix D were analyzed by only a few laboratories and, in some cases, by just one laboratory. Caution should be exercised in the use of this data.

Table 3 – Analytical information for the following elements/constituents available in Appendix D

F	Ba
Cl	Cr
Co	Cu
C	Mn
U–HNO_3 insoluble	Pb
Ag	Sr
Al	Th
Be	Zn

Anomalies that were immediately apparent upon inspection of the data are shown in Table 4. Some laboratories tried to use neutron activation analysis, but it was thought that the production of fission products caused by the high concentration of uranium in CUP-2 produced interference in the gamma-ray measurements. A conclusion easily reached from the data is that analyte line selection when using ICP or DCP is critical, as very high error can be caused by interference from the rich uranium spectrum.

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4. Steger, H.F. "A re-assessment of the criterion for certifiability in CCRMP"; *Geostandards Newsletter* 6:1:17-23; 1982.

5. Sutarno, R. and Steger, H.F. "The use of certified reference materials in the verification of analytical data and methods"; *Talanta* 32:6:439-445; 1985.

Table 4 – Anomalies in analytical data

K	Two sets of results are higher than the bulk of the other results by a factor of 3. Suspect that these outliers were obtained by neutron activation analysis.
Mo	Most results obtained by AA. One set by ICAP high by 100%. One set by AA high by 300%.
Na	One low outlier obtained by AA.
B	One high outlier obtained by ICP.
Zr	One high outlier obtained by DCP.
V	Two sets of results higher than the majority by a factor of two. Both were obtained by ICP.
Si	Two extreme outliers, one high and one low. High result was obtained by gravimetry, and the low result was obtained by emission spectroscopy.

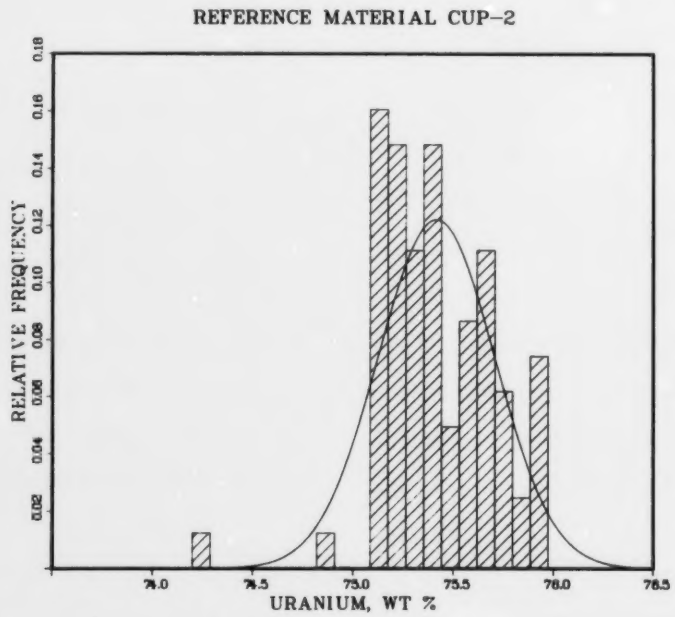

Fig. 1 – Histogram of uranium results

APPENDIX A

CONFIRMATION OF HOMOGENEITY

CONFIRMATION OF HOMOGENEITY

The homogeneity of CUP-2 was assessed with respect to uranium, arsenic and iron by X-ray fluorescence analysis performed at CANMET. This was done by counting triplicate aliquots from 15 bottles selected from a stock of 450 bottles. The stock was divided into lots of 50 bottles. The code number of the first bottle was selected at random out of the first lot. The code number of the remaining bottles selected was determined by the code number of the preceding bottle plus 9. The results are shown in Tables A5, A6 and A7.

A Spex XRF plastic cup was completely filled with the material (approximately 5 g) and a Mylar film was used to cover the cell, which was in the inverted position during the XRF measurement.

XRF MEASUREMENTS

Molybdenum target X-ray tube operated at 50 kV 40 mA

LiF_{200} crystal

UL_{α} at 30.69°

AsK_{α} 33.94°

FeK_{α} 57.49°

A one-way ANOVA technique (2) was used to assess the homogeneity wherein the ratio of the between-bottle to within-bottle mean squares are compared with the *F* statistic at the 95% level of probability. The null hypothesis of no difference between bottles was accepted in each of the three cases.

Table A5 – Confirmation of homogeneity of uranium

XRF counts per second		
13317.	13452.	13606.
13550.	13665.	13340.
13550.	13515.	13630.
13615.	13660.	13658.
13551.	13600.	13632.
13616.	13314.	13494.
13619.	13494.	13632.
13614.	13663.	13635.
13609.	13386.	13645.
13750.	13666.	13438.
13494.	13463.	13309.
13514.	13420.	13312.
13673.	13552.	13511.
13428.	13687.	13360.
13572.	13692.	13777.

Analysis of variance table			
Source of variation	Degrees of freedom	Sum of squares	Mean squares
Between sets	14	.28538711E+06	.20384794E+05
Within sets	30	.39320800E+06	.13106933E+05
Total	44	.67859511E+06	
Calculated F statistic:	1.555		
$F_{0.95}$ (14,30):	2.037		
Null hypothesis of no difference between bottles is accepted for uranium.			

Table A6 – Confirmation of homogeneity of arsenic

XRF counts per second		
436.	434.	449.
445.	447.	454.
441.	451.	434.
458.	439.	454.
448.	444.	449.
438.	425.	453.
437.	440.	441.
435.	429.	453.
420.	450.	454.
460.	448.	450.
435.	443.	452.
448.	448.	435.
447	453.	443.
447.	450.	444.
444.	461.	454.

Analysis of variance table			
Source of variation	Degrees of freedom	Sum of squares	Mean squares
Between sets	14	.10297778E+04	.73555556E+02
Within sets	30	.24966667E+04	.83222222E+02
Total	44	.35264444E+04	
Calculated F statistic:	0.884		
$F_{0.95}$ (14,30):	2.037		
Null hypothesis of no difference between bottles is accepted for arsenic.			

Table A7 – Confirmation of homogeneity of iron

XRF counts per second		
728.	695.	708.
714.	720.	710.
724.	735.	741.
709.	714.	742.
711.	713.	721.
726.	711.	731.
730.	716.	731.
708.	741.	723.
736.	703.	709.
728.	720.	712.
725.	711.	729.
714.	691.	712.
711.	707.	715.
712.	715.	706.
730.	717.	712.

Analysis of variance table			
Source of variation	Degrees of freedom	Sum of squares	Mean squares
Between sets	14	.21539111E+04	.15385079E+03
Within sets	30	.38400000E+04	.12800000E+03
Total	44	.59939111E+04	
Calculated F statistic:	1.202		
$F_{0.95}$ (14,30):	2.037		
Null hypothesis of no difference between bottles is accepted for iron.			

APPENDIX B

PARTICIPATING LABORATORIES

PARTICIPATING LABORATORIES

Allied Corporation
Metropolis, Illinois, U.S.A.
T.L. Hulsey, G. Gray, C.A. Gimm, J.C. Kern, E.L. Caldwell, W.M. Davis

*Barringer Magenta
Rexdale, Ontario
A. Lip[illegible]i, M. Muneswar

*Bondar–Clegg & Co. Ltd.
Ottawa, Ontario
P. Haulena

CANMET
Energy, Mines and Resources Canada
Ottawa, Ontario
E. Cyfracki, J.L. Dalton, W.S. Bowman

Cluff Mining
Saskatoon, Saskatchewan
C. Lee

Earth Sciences Extraction Co.
Calgary, Alberta
R. Blackmore

Eldorado Resources Ltd.
Ottawa, Ontario
G.A. Dunlop

Eldorado Resources Ltd.
Blind River, Ontario
T.P. Smith

Denison Mines Limited
Elliot Lake, Ontario
D.–H. Kim

*Kamloops Research and Assay Laboratory Ltd.
Kamloops, B.C.
D.A. Blundell

Key Lake Mining Corporation
Saskatoon, Saskatchewan
K. Ko

*Nuclear Activation Services Ltd.
Hamilton, Ontario

Pacific North West Laboratories
Richland, Washington, U.S.A.
M.C. Burt, J.J. McCown

Ranger Uranium Mines Pty. Ltd.
Jabiru, N.T., Australia
H. Russell

Rio Algom Ltd.
Elliot Lake, Ontario
B. Donahue, A. Enright, J. Nipius

*Saskatchewan Research Council
Saskatoon, Saskatchewan
W. Yuen, L. MacDonald

*X–ray Assay Laboratories Ltd.
Don Mills, Ontario
J. Opdebeeck

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*Supply and Services Canada Contract Serial No. 23440–6–9051/OX–SQ, 12–08–86

APPENDIX C

SUMMARY OF STATISTICAL PARAMETERS

SUMMARY OF STATISTICAL PARAMETERS

Data generated by the interlaboratory program were analyzed statistically. Appendix C lists the statistical parameters for the certified and recommended values. The following explanation is given for symbols shown.

No. of Labs	Total number of laboratories providing data for a particular analyte.
Total N	Total number of determinations available for a particular analyte.
Mean	The arithmetic mean, after exclusion of outliers, of all the data for a particular analyte.
95% Conf Int Low High	The confidence interval calculated from the data using the t distribution.
Median	The value exactly in the middle of all analytical data for an analyte.
Spread, %	The difference between the high and low confidence interval divided by the mean.
Average SD (σ_A)	The average standard deviation of all the data for an analyte.
CC and RP	Criterion for certification and rejection percentage as explained earlier in the main body of the report.
Within-lab SD, (S_{rc})	The within-laboratory component of the standard deviation, σ.
Between-lab SD, (S_{Lc})	The between-laboratory component of the standard deviation, ω.

Table C8 – Statistics for CUP–2

	Uranium wt %	Moisture wt %	Sulphur wt %	Silicon wt %	Titanium wt %	Calcium wt %
No. of Labs	13	12	11	12	9	12
Total N	81	62	55	68	50	64
Mean	75.42	2.94	0.80	0.17	0.019	0.62
95% Conf Int						
Low	75.25	2.76	0.75	0.11	0.016	0.59
High	75.59	3.12	0.86	0.23	0.022	0.66
Median	75.40	2.96	0.82	0.15	0.017	0.64
Spread, %	0.45	12.6	13.5	70.3	34.8	10.9
Average SD	0.12	0.06	0.02	0.01	0.001	0.01
CC	2.4	2.8	2.3	2.7	2.9	2.7
RP	0.0	30.8	25.0	46.2	20.0	30.8
Within–lab SD	0.16	0.07	0.02	0.02	0.002	0.02
Between–lab SD	0.25	0.27	0.08	0.09	0.004	0.05

Table C8 (cont'd)

	Vanadium wt %	Arsenic wt %	Zirconium wt %	Boron µg/g	Iron wt %	Nickel µg/g
No. of Labs	9	9	4	7	13	9
Total N	44	50	25	36	69	50
Mean	0.066	0.035	0.044	51.	0.311	29.
95% Conf Int						
Low	0.062	0.032	0.032	38.	0.295	22.
High	0.071	0.038	0.056	64.	0.326	36.
Median	0.066	0.035	0.045	50.	0.317	25.
Spread, %	14.4	15.7	54.1	50.7	9.9	49.5
Average SD	0.002	0.001	0.002	4.	0.007	2.
CC	2.9	2.5	0.6	2.5	2.5	3.0
RP	36.4	33.3	50.0	25.0	35.7	20.0
Within-lab SD	0.002	0.001	0.002	5.	0.007	1.
Between-lab SD	0.006	0.003	0.007	14.	0.025	9.

Table C8 (cont'd)

	Sodium wt %	Molybdenum wt %	Magnesium wt %	Potassium wt %	Phosphorus wt %
No. of Labs	13	9	13	11	4
Total N	69	44	69	58	20
Mean	0.459	0.069	0.229	0.109	0.030
95% Conf Int					
Low	0.433	0.064	0.213	0.102	0.021
High	0.485	0.075	0.245	0.116	0.040
Median	0.445	0.070	0.234	0.110	0.030
Spread, %	11.3	15.7	14.0	13.0	63.6
Average SD	0.017	0.002	0.009	0.003	0.002
CC	2.4	2.7	2.6	2.9	2.8
RP	7.1	27.3	7.7	21.4	0.0
Within-lab SD	0.023	0.002	0.011	0.004	0.003
Between-lab SD	0.041	0.007	0.025	0.010	0.006

APPENDIX D

ANALYTICAL DATA RECEIVED

ANALYTICAL DATA RECEIVED

Analytical results, means, and standard deviations for uranium in CUP-2

Laboratory	Method[+]	Uranium, Wt % (dry basis)					Mean	S.D.
LAB- 1	10	75.41	75.43	75.29	75.29		75.35	0.08
LAB- 2	4	75.62	75.79	75.62	75.80	75.67	75.70	0.09
LAB- 3	4	75.12	75.19	75.35	75.20	75.23	75.24	0.12
		75.50	75.19	75.14	75.17	75.28		
		75.41	75.13					
LAB- 4	4	75.6	75.2	74.9	75.4	74.2	75.06	0.55
LAB- 5	10	75.356	75.338	75.466	75.310	75.334	75.36	0.06
LAB- 6	10	75.13	75.10	75.11	75.10	75.18	75.14	0.04
		75.19	75.21	75.17	75.11	75.10		
LAB- 8*	4	74.3	74.1	72.5	75.0	74.8	74.14	0.99
LAB- 9	10	75.766	75.891	75.783	75.917	75.919	75.86	0.07
LAB-10	10	75.23	75.17	75.23	75.19	75.17	75.20	0.03
LAB-11	10	75.58	75.64	75.71	75.42	75.63	75.60	0.11
LAB-12	10	75.974	75.882	75.936	75.97	75.78	75.91	0.08
LAB-13	10	75.44	75.41	75.39	75.36	75.39	75.40	0.03
LAB-13	4	75.50	75.54	75.29	75.24	75.42	75.40	0.13
LAB-14	10	75.65	75.55	75.53	75.67	75.60	75.57	0.12
		75.57	75.62	75.67	75.27	75.61		

Analytical results, means, and standard deviations for moisture in CUP-2

Laboratory	Method	Moisture, Wt %					Mean	S.D.
LAB- 1	13	2.90	2.93				2.92	0.02
LAB- 2	13	2.936	3.132	3.144	3.100	3.083	3.08	0.08
LAB- 3	13	3.10	3.11	3.23	3.14	3.28	3.17	0.07
		3.17						
LAB- 4	13	3.02	2.98	2.94	2.98	3.04	2.99	0.04
LAB- 5	13	3.150	3.166	3.104	3.055	2.986	3.09	0.07
LAB- 6	13	2.71	2.68	2.67	2.58	2.58	2.64	0.06
LAB- 8	13	2.6	2.5	2.5	2.6	2.5	2.54	0.05
LAB- 9	13	3.491	3.465	3.492	3.376	3.492	3.46	0.05
LAB-10	13	2.70	2.66	2.59	2.64	2.59	2.64	0.05
LAB-11*	13	2.17	2.08	2.31	1.88	2.34	2.16	0.19
LAB-12	13	3.183	3.129	3.243	3.288	3.056	3.18	0.09
LAB-13	13	3.01	3.01	2.94	2.89		2.96	0.06
LAB-14	13	2.83	2.81	2.84	2.82	2.86	2.75	0.10
		2.64	2.63	2.62	2.64	2.76		

[+] See last page of Table for method details.
* Declared outlying set.

Analytical results, means, and standard deviations for sulphur in CUP-2

Laboratory	Method	Sulphur, Wt %					Mean	S.D.
LAB- 2	14	0.858	0.859	0.844	0.847	0.852	0.85	0.007
LAB- 4	11	0.81	0.80	0.86	0.82	0.81	0.82	0.024
LAB- 5	13	0.826	0.812	0.833	0.835	0.823	0.83	0.009
LAB- 6*	11	0.515	0.550	0.560	0.465	0.665	0.55	0.074
LAB- 8	13	0.83	0.87	0.82	0.88	0.84	0.85	0.026
LAB- 9	13	0.759	0.761	0.784	0.843	0.793	0.79	0.034
LAB-10	13	0.82	0.83	0.79	0.84	0.896	0.84	0.039
LAB-11	13	0.62	0.61	0.61	0.63	0.64	0.62	0.013
LAB-12	12	0.923	0.924	0.931	0.925	0.916	0.92	0.005
LAB-13	13	0.85	0.85	0.84	0.84	0.84	0.84	0.006
LAB-15	12	0.69	0.76	0.75	0.73	0.73	0.73	0.027
LAB-16*	11	1.2					1.20	-----
LAB-17	13	0.74	0.73	0.73	0.73	0.76	0.74	0.013

Analytical results, means, and standard deviations for silicon in CUP-2

Laboratory	Method	Silicon, Wt %					Mean	S.D.
LAB- 1	13	0.154	0.154	0.154	0.149		0.15	0.003
LAB- 2	4	0.211	0.213	0.214	0.200	0.205	0.21	0.006
LAB- 4	3	0.310	0.308	0.308	0.308	0.303	0.31	0.003
LAB- 5	3	0.04	0.03	0.03	0.03	0.03	0.03	0.005
LAB- 6	1	0.1425	0.1461	0.1418	0.1448	0.1440	0.13	0.018
		0.1090	0.1106	0.1091	0.1113	0.1115		
LAB- 8	2	0.0993	0.0978	0.0921	0.1040	0.1010	0.10	0.004
LAB-10*	13	0.685	0.542	0.557	0.682	0.434	0.58	0.106
LAB-11	13	0.35	0.35	0.39	0.36	0.35	0.36	0.017
LAB-12	5	0.05	0.06	0.10	0.09	0.08	0.08	0.021
LAB-13	13	0.19	0.18	0.19	0.19	0.20	0.19	0.007
LAB-14	13	0.15	0.15	0.09	0.11	0.14	0.14	0.027
		0.16	0.16	0.17	0.17			
LAB-15	1	0.203	0.185	0.207	0.218	0.184	0.20	0.015
LAB-16*	3	0.21					0.21	-----
LAB-17	13	0.168	0.154	0.149	0.154	0.154	0.16	0.007

* Declared outlying set.

Analytical results, means, and standard deviations for titanium in CUP-2

Laboratory	Method	Titanium, Wt %					Mean	S.D.
LAB- 2	4	0.0181	0.0165	0.0173	0.0177	0.0175	0.017	0.0006
LAB- 3	3	0.0137	0.0136	0.0120	0.0152	0.0137	0.014	0.0011
LAB- 5	3	0.025	0.025	0.021	0.019	0.021	0.022	0.0027
LAB- 6	1	0.0174	0.0175	0.0170	0.0168	0.0175	0.017	0.0003
		0.0171	0.0169	0.0174	0.0170	0.0169		
LAB- 8	2	0.0232	0.026	0.0261	0.0274	0.0268	0.026	0.0016
LAB-11*	2	0.036	0.036	0.036	0.038	0.038	0.037	0.0011
LAB-12	3	0.0152	0.0156	0.0153	0.0156	0.0152	0.015	0.0002
LAB-13	2	0.022	0.021	0.018	0.015	0.025	0.020	0.0038
LAB-15	1	0.0171	0.0163	0.0166	0.0162	0.0169	0.017	0.0004
LAB-16*	3	0.02					0.020	-----
LAB-17	3	0.025	0.025	0.025	0.025	0.025	0.025	0.0000

Analytical results, means, and standard deviations for calcium in CUP-2

Laboratory	Method	Calcium, Wt %					Mean	S.D.
LAB- 1	3	0.65	0.65	0.64	0.64		0.645	0.006
LAB- 2	4	0.648	0.647	0.635	0.649	0.642	0.644	0.006
LAB- 3	3	0.6220	0.6492	0.6214	0.6248	0.6335	0.630	0.012
LAB- 4	3	0.668	0.667	0.670	0.670	0.692	0.673	0.011
LAB- 5	3	0.634	0.654	0.674	0.614	0.634	0.642	0.023
LAB- 6	1	0.6696	0.6735	0.6846	0.6683	0.6680	0.667	0.012
		0.6805	0.6674	0.6467	0.6552	0.6531		
LAB- 8	2	0.666	0.677	0.678	0.693	0.690	0.681	0.011
LAB-10	3	0.661	0.637	0.627	0.648	0.664	0.647	0.016
LAB-11	3	0.59	0.58	0.55	0.55	0.54	0.562	0.022
LAB-12	3	0.5574	0.5631	0.5567	0.5644	0.5592	0.560	0.003
LAB-13	3	0.54	0.54	0.58	0.52	0.55	0.546	0.022
LAB-15	1	0.523	0.534	0.571	0.564	0.530	0.544	0.022
LAB-16*	3	0.80					0.800	-----
LAB-17*	3	1.02	1.13	1.09	1.03	1.03	1.060	0.048

* Declared outlying set.

Analytical results, means, and standard deviations for vanadium in CUP-2

Laboratory	Method	Vanadium, Wt %					Mean	S.D.
LAB- 1	3	0.06	0.06	0.06	0.06		0.060	0.000
LAB- 2	4	0.0674	0.0675	0.0664	0.0676	0.0692	0.068	0.001
LAB- 3	3	0.0628	0.0633	0.0660	0.0641	0.0657	0.064	0.001
LAB- 4	3	0.0697	0.0695	0.0689	0.0690	0.0689	0.069	0.000
LAB- 5	3	0.076	0.081	0.076	0.081	0.081	0.079	0.003
LAB- 6*	1	0.1367	0.1383	0.1352	0.1389	0.1403	0.138	0.002
		0.1391	0.1409	0.1367	0.1355	0.1408		
LAB- 8	2	0.0712	0.0701	0.0717	0.0753	0.0715	0.072	0.002
LAB-11	3	0.064	0.060	0.064	0.062	0.066	0.063	0.002
LAB-12	3	0.0592	0.0588	0.0589	0.0594	0.0587	0.059	0.000
LAB-13	2	0.066	0.061	0.067	0.063	0.064	0.064	0.002
LAB-15*	1	0.125	0.111	0.116	0.130	0.127	0.122	0.008
LAB-16*	3	0.08					0.080	-----

Analytical results, means, and standard deviations for arsenic in CUP-2

Laboratory	Method	Arsenic, Wt %					Mean	S.D.
LAB- 4	15	0.0333	0.0336	0.0334	0.0339	0.0339	0.0336	0.0003
LAB- 6	1	0.0349	0.0385	0.0355	0.0395	0.0385	0.0372	0.0015
		0.0367	0.0368	0.0364	0.0360	0.0387		
LAB- 8	6	0.0404	0.0402	0.0403	0.0404	0.0402	0.0403	0.0001
LAB-10	15	0.0371	0.0376	0.0370	0.0371	0.0371	0.0372	0.0002
LAB-11	9	0.0346	0.0336	0.0332	0.0344	0.0344	0.0340	0.0006
LAB-12	3	0.0286	0.0296	0.0288	0.0292	0.0295	0.0291	0.0004
LAB-13	2	0.030	0.028	0.033	0.032	0.032	0.0310	0.0020
LAB-15	3	0.0375	0.0375	0.0375	0.0375	0.0375	0.0375	0.0000
LAB-17	3	0.034	0.034	0.034	0.031	0.034	0.0334	0.0013

* Declared outlying set.

Analytical results, means, and standard deviations for zirconium in CUP-2

Laboratory	Method	Zirconium, Wt %					Mean	S.D.
LAB- 6	1	0.0446	0.0450	0.0462	0.0455	0.0450	0.0446	0.0010
		0.0444	0.0436	0.0448	0.0435	0.0429		
LAB- 8	2	0.0525	0.0531	0.0546	0.0562	0.0551	0.0543	0.0015
LAB-12	3	0.0358	0.0354	0.0364	0.0339	0.0348	0.0353	0.0010
LAB-13	2	0.012	0.008	0.013	0.009		0.0105	0.0024
LAB-15	1	0.0406	0.0464	0.0454	0.0427	0.0408	0.0432	0.0026
LAB-16*	2	0.19					0.1900	------

Analytical results, means, and standard deviations for boron in CUP-2

Laboratory	Method	Boron, µg/g					Mean	S.D.
LAB- 3	5	36.	35.	37.	38.	35.	36.2	1.2
		36.						
LAB- 6*	1	216.	213.	208.	203.	207.	209.4	3.7
		208.	208.	210.	213.	208.		
LAB- 7	7	50.	51.	53.	49.	54.	51.4	2.1
LAB- 8	2	63.	57.	63.	65.	65.	62.6	3.3
LAB-11	9	80.	80.	70.	70.	70.	74.0	5.5
LAB-12	5	50.	50.	30.	30.	30.	38.0	11.0
LAB-13	2	40.	40.	40.	40.	50.	42.0	4.5
LAB-15	1	53.	57.	54.	59.	50.	54.6	3.5

* Declared outlying set.

Analytical results, means, and standard deviations for iron in CUP-2

Laboratory	Method	Iron, Wt %					Mean	S.D.
LAB- 1	3	0.32	0.32	0.33	0.32		0.323	0.005
LAB- 2	4	0.333	0.334	0.335	0.341	0.333	0.335	0.003
LAB- 3	3	0.3316	0.3281	0.3527	0.3275	0.3255	0.333	0.011
LAB- 4*	3	0.363	0.375	0.375	0.372	0.375	0.372	0.005
LAB- 5	3	0.276	0.292	0.276	0.280	0.276	0.280	0.007
LAB- 6	1	0.3137	0.3229	0.3213	0.3281	0.3154	0.322	0.005
		0.3216	0.3263	0.3302	0.3233	0.3171		
LAB- 8	2	0.338	0.347	0.350	0.351	0.341	0.345	0.006
LAB- 9	9	0.285	0.277	0.279	0.278	0.278	0.279	0.003
LAB-10	3	0.352	0.325	0.329	0.348	0.343	0.339	0.012
LAB-11	3	0.32	0.32	0.32	0.30	0.31	0.314	0.009
LAB-12	3	0.3041	0.3035	0.2992	0.2997	0.3036	0.302	0.002
LAB-13	3	0.28	0.29	0.27	0.28	0.29	0.282	0.008
LAB-15	1	0.265	0.272	0.274	0.289	0.276	0.275	0.009
LAB-16*	9	0.46					0.460	-----
LAB-17	3	0.30	0.30	0.30	0.29	0.30	0.298	0.005

Analytical results, means, and standard deviations for nickel in CUP-2

Laboratory	Method	Nickel, µg/g					Mean	S.D.
LAB- 3	3	38.	36.	33.	33.	32.	34.6	2.7
LAB- 4	3	23.	25.	24.	23.	21.	23.2	1.5
LAB- 5	3	52.	52.	48.	48.	52.	50.4	2.2
LAB- 6	1	24.	23.	23.	24.	24.	23.4	0.5
		24.	23.	23.	23.	23.		
LAB- 8*	2	72.	71.	69.	80.	76.	73.6	4.4
LAB-11	3	20.	20.	20.	20.	20.	20.0	0.0
LAB-12	3	25.	25.	25.	25.	25.	25.0	0.0
LAB-13	2	30.	30.	30.	30.	30.	30.0	0.0
LAB-15	1	25.	28.	25.	28.	25.	26.2	1.6
LAB-17	3	30.	30.	30.	30.	30.	30.0	0.0

* Declared outlying set.

Analytical results, means, and standard deviations for sodium in CUP-2

Laboratory	Method	Sodium, Wt %					Mean	S.D.
LAB- 1	3	0.47	0.46	0.46	0.44		0.458	0.013
LAB- 2	3	0.388	0.392	0.399	0.399	0.392	0.394	0.005
LAB- 3	3	0.4670	0.4635	0.4642	0.4604	0.4624	0.464	0.002
LAB- 4	3	0.443	0.434	0.475	0.423	0.436	0.442	0.020
LAB- 5	3	0.530	0.540	0.530	0.530	0.550	0.536	0.009
LAB- 6	1	0.4244	0.4273	0.4096	0.4082	0.4323	0.422	0.008
		0.4312	0.4245	0.4195	0.4179	0.4260		
LAB- 7	8	0.56	0.48	0.51	0.54	0.56	0.530	0.035
LAB- 8	3	0.447	0.448	0.437	0.436	0.444	0.442	0.006
LAB-10	3	0.421	0.441	0.473	0.426	0.445	0.441	0.020
LAB-11*	3	0.28	0.26	0.26	0.26	0.28	0.268	0.011
LAB-12	3	0.4995	0.4953	0.4793	0.4813	0.4859	0.488	0.009
LAB-13	2	0.47	0.56	0.43	0.39	0.42	0.454	0.066
LAB-15	1	0.48	0.49	0.48	0.53	0.51	0.498	0.022
LAB-17	3	0.44	0.43	0.43	0.41	0.44	0.430	0.012

Analytical results, means, and standard deviations for molybdenum in CUP-2

Laboratory	Method	Molybdenum, Wt %					Mean	S.D.
LAB- 1	3	0.07	0.08	0.07	0.07		0.073	0.005
LAB- 3	3	0.0729	0.0724	0.0732	0.0733	0.0712	0.073	0.001
LAB- 4	3	0.0782	0.0786	0.0770	0.0748	0.0755	0.077	0.002
LAB- 5*	3	0.240	0.220	0.250	0.230	0.240	0.236	0.011
LAB- 6	1	0.0656	0.0683	0.0667	0.0688	0.0687	0.068	0.001
LAB- 8	2	0.0697	0.0704	0.0688	0.0725	0.0684	0.070	0.002
LAB-11	3	0.060	0.057	0.056	0.057	0.054	0.057	0.002
LAB-12	3	0.0570	0.0613	0.0572	0.0617	0.0592	0.059	0.002
LAB-13	2	0.074	0.068	0.074	0.069	0.069	0.071	0.003
LAB-15*	1	0.1820	0.1680	0.1610	0.1640	0.1650	0.168	0.008
LAB-16*	3	0.12					0.120	-----
LAB-17	3	0.076	0.077	0.077	0.076	0.076	0.076	0.001

* Declared outlying set.

Analytical results, means, and standard deviations for magnesium in CUP-2

Laboratory	Method	Magnesium, Wt %					Mean	S.D.
LAB- 1	3	0.25	0.22	0.24	0.24		0.238	0.013
LAB- 2	3	0.252	0.249	0.244	0.249	0.248	0.248	0.003
LAB- 3	3	0.2331	0.2301	0.2306	0.2270	0.2291	0.230	0.002
LAB- 4	3	0.230	0.229	0.229	0.233	0.228	0.230	0.002
LAB- 5	3	0.210	0.206	0.220	0.222	0.228	0.217	0.009
LAB- 6	1	0.2432	0.2397	0.2462	0.2454	0.2426	0.240	0.004
		0.2350	0.2375	0.2340	0.2370	0.2427		
LAB- 8	2	0.253	0.262	0.265	0.264	0.255	0.260	0.005
LAB-10	3	0.230	0.234	0.263	0.229	0.232	0.238	0.014
LAB-11	3	0.248	0.248	0.251	0.242	0.246	0.247	0.003
LAB-12	3	0.1568	0.1819	0.1640	0.1873	0.1823	0.175	0.013
LAB-13	2	0.20	0.21	0.16	0.14	0.21	0.184	0.032
LAB-15	1	0.205	0.209	0.211	0.213	0.202	0.208	0.005
LAB-16*	3	0.34					0.340	-----
LAB-17	3	0.26	0.25	0.25	0.26	0.26	0.256	0.006

Analytical results, means, and standard deviations for potassium in CUP-2

Laboratory	Method	Potassium, Wt %					Mean	S.D.
LAB- 1	3	0.11	0.10	0.10	0.10		0.103	0.005
LAB- 2	3	0.098	0.097	0.097	0.097	0.097	0.097	0.000
LAB- 3	3	0.1167	0.1159	0.1158	0.1137	0.1140	0.115	0.001
LAB- 4	3	0.108	0.128**	0.110	0.109	0.109	0.113	0.009
LAB- 5	3	0.092	0.093	0.093	0.094	0.091	0.093	0.001
LAB- 6	1	0.1153	0.1187	0.1214	0.1153	0.1214	0.119	0.005
		0.1168	0.1302	0.1149	0.1194	0.1162		
LAB- 7*	8	0.38	0.38	0.38	0.38	0.38	0.380	0.000
LAB- 8	3	0.103	0.100	0.102	0.106	0.101	0.102	0.002
LAB-10	3	0.104	0.110	0.110	0.107	0.109	0.108	0.003
LAB-11*	3	0.06	0.06	0.06	0.06	0.06	0.060	0.000
LAB-12	3	0.0981	0.0992	0.0989	0.1004	0.0984	0.099	0.001
LAB-13	2	0.13	0.12	0.13	0.12	0.12	0.124	0.006
LAB-15*	1	0.37	0.37	0.37	0.39	0.37	0.374	0.009
LAB-17	3	0.12	0.12	0.11	0.11	0.11	0.114	0.006

* Declared outlying set.
** Declared outlying value.

Analytical results, means, and standard deviations for fluorine in CUP-2

Laboratory	Method	Fluorine, Wt %					Mean	S.D.
LAB- 8	16	0.0175	0.0165	0.0150	0.0173	0.0155	0.016	0.001
LAB-11	16	0.012	0.012	0.011	0.012	0.014	0.012	0.001
LAB-16	16	0.01					0.010	-----

Analytical results, means, and standard deviations for chlorine in CUP-2

Laboratory	Method	Chlorine, Wt %					Mean	S.D.
LAB- 8	16	0.0336	0.0338	0.0332	0.0327	0.0340	0.034	0.001
LAB-16	9	0.02					0.020	-----

Analytical results, means, and standard deviations for carbonate in CUP-2

Laboratory	Method	Carbonate, Wt %					Mean	S.D.
LAB- 8	13	0.18	0.19	0.20	0.19	0.20	0.19	0.01
LAB-16	13	0.25					0.25	----

Analytical results, means, and standard deviations for carbon in CUP-2

Laboratory	Method	Carbon, Wt %					Mean	S.D.
LAB- 6	11	0.060	0.060	0.070	0.065	0.065	0.064	0.004

Analytical results, means, and standard deviations for uranium - HNO_3 insoluble in CUP-2

Laboratory	Method	Uranium, µg/g					Mean	S.D.
LAB-17	9	34.	31.	29.	30.	30.	30.8	1.9

Analytical results, means, and standard deviations for silver in CUP-2

Laboratory	Method	Silver, µg/g					Mean	S.D.
LAB-15	1	101.	129.	130.	169.	158.	137.	27.

Analytical results, means, and standard deviations for aluminum in CUP-2

Laboratory	Method	Aluminum, Wt %					Mean	S.D.
LAB- 1	3	0.29	0.23	0.23	0.23		0.245	0.030
LAB-15	1	0.226	0.232	0.242	0.262	0.243	0.241	0.014

Analytical results, means, and standard deviations for beryllium in CUP-2

Laboratory	Method	Beryllium, µg/g					Mean	S.D.
LAB-15	1	1.24	1.63	1.06	1.55	1.02	1.30	0.28

Analytical results, means, and standard deviations for barium in CUP-2

Laboratory	Method	Barium, µg/g					Mean	S.D.
LAB-15	1	196.	199.	196.	206.	195.	198.	4.5

Analytical results, means, and standard deviations for chromium in CUP-2

Laboratory	Method	Chromium, Wt %					Mean	S.D.
LAB- 6	1	0.0019	0.0017	0.0016	0.0017	0.0017	0.0016	0.0001
		0.0015	0.0015	0.0015	0.0015	0.0014		
LAB-15	1	0.0215	0.0254	0.0239	0.0243	0.0255	0.0241	0.0016

Analytical results, means, and standard deviations for copper in CUP-2

Laboratory	Method	Copper, µg/g					Mean	S.D.
LAB- 6	1	42.	38.	34.	32.	31.	31.	5.6
		27.	27.	26.	26.	26.		
LAB-15	1	22.	22.	22.	22.	22.	22.	0.0

Analytical results, means, and standard deviations for manganese in CUP-2

Laboratory	Method	Manganese, µg/g					Mean	S.D.
LAB- 6	1	103.	101.	104.	104.	102.	103.	1.1
		101.	103.	103.	103.	102.		
LAB-15	1	109.	95.	99.	93.	93.	98.	6.7

Analytical results, means, and standard deviations for phosphorus in CUP-2

Laboratory	Method	Phosphorus, Wt %					Mean	S.D.
LAB- 8	2	0.0270	0.0286	0.0285	0.0298	0.0292	0.029	0.001
LAB- 9	13	0.0236	0.0237	0.0216	0.0248	0.0222	0.023	0.001
LAB-11	9	0.034	0.033	0.031	0.031	0.033	0.032	0.001
LAB-15	1	0.034	0.032	0.044	0.037	0.041	0.038	0.005
LAB-16*	9	0.01					0.010	-----

Analytical results, means, and standard deviations for lead in CUP-2

Laboratory	Method	Lead, Wt %					Mean	S.D.
LAB-15	1	0.0255	0.0265	0.0245	0.0255	0.0265	0.0257	0.0008

Analytical results, means, and standard deviations for strontium in CUP-2

Laboratory	Method	Strontium, µg/g					Mean	S.D.
LAB-15	1	46.	44.	46.	50.	48.	47.	2.3

Analytical results, means, and standard deviations for thorium in CUP-2

Laboratory	Method	Thorium, Wt %					Mean	S.D.
LAB- 8	17	0.170	0.176	0.172	0.167	0.171	0.171	0.003
LAB-15	1	0.269	0.281	0.293	0.294	0.301	0.288	0.013
LAB-16	9	0.20					0.200	-----

* Declared outlying set.

Analytical results, means, and standard deviations for zinc in CUP-2

Laboratory	Method	Zinc, µg/g					Mean	S.D.
LAB-15	1	36.	36.	36.	37.	36.	36.2	0.4

Method Codes

1. ICP
2. DCP
3. AA
4. XRF
5. OES
6. FAA
7. Prompt gamma-ray
8. NAA
9. Spectrophotometry
10. Redox Titrimetry
11. Combustion & IR
12. Combustion & Iodometry
13. Gravimetry
14. Turbidimetry
15. Hydride AA
16. Ion Specific Electrode
17. ICPMS

Uranium results are on a dry basis. All others are on an "as received" basis. Laboratories 6,8,12,& 13 extracted the uranium prior to the metals determinations.